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14. ABSTRACT

At the onset of this Project AMSC used an R&D scale to demonstrate the AMSC 2G HTS process in 10 m long, 10 mm wide 2G conductors which showed 250 A end-to-end critical currents Ic at 77 K. Standard deviation of Ic in these wires was low and index values n (V~In) were quite high, ranging from 31 to 37. For a more cost-effective full scale 2G HTS production, a processing width of 100 mm is envisioned, with a processing length of >1000m. Only at the very end of the process, after coating of the Ag cap layer, is the conductor slit to final conductor width which is around 4 mm. To bridge this gap between the 10 and 100 mm process width, the 2G HTS development used an in-between process width of 40 mm. During the Phase II the process width remained at 4 cm, and length was around 130 m. At the end of the Phase II the length was extended to 500 m.

15. SUBJECT TERMS

YBCO Coated Conductor, AC losses, RABiTS, Superconductor, Maganetic Properties

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Air Force Office of Scientific Research
Phase II STTR

Contract Number FA9550-05-C-0025

"YBCO COATED CONDUCTORS WITH REDUCED AC LOSSES"

FINAL TECHNICAL REPORT

Prepared by

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The Applied Superconductivity Center
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1. INTRODUCTION

Second Generation High Temperature Superconductors (2G HTS) are seen as the major candidate for military high energy density HTS application such as turbo-generators and gyrotron magnets. The major reason is the enhanced in-field performance at 50-65 K and the proven effectiveness of artificial flux pinning. One key advantage is the possibility of achieving low AC losses in a variety of applications, including cables, transformers, current limiters and the stators of rotating equipment. Low AC-loss in 2G HTS requires wire components with low magnetism, and an YBCO layer with low transport and low hysteretic losses in an alternating magnetic field. The latter loss type requires a suitable filamentization technique to split the YBCO layer in parallel, narrow filaments, and a method for filament transposition to reduce coupling losses. These two subjects for ac loss reduction (low loss substrates and a novel approach to filament transposition) have been major topics in this Phase II Project.

At the onset of this Project AMSC used an R&D scale to demonstrate the AMSC 2G HTS process in 10 m long, 10 mm wide 2G conductors which showed 250 A end-to-end critical currents I_c at 77 K. Standard deviation of I_c in these wires was low and index values n ($V \sim I^n$) were quite high, ranging from 31 to 37. This R&D 10 mm process width is not a cost-effective approach to manufacturing. For a full scale 2G HTS production a processing width of 100 mm is envisioned, with a processing length of >1000m. Only at the very end of the process, after coating of the Ag cap layer, is the conductor slit to final conductor width which is around 4 mm. To bridge this gap between the 10 and 100 mm process width, the 2G HTS development used an in-between process width of 40 mm. During the Phase II the process width remained at 4 cm, and length was around 130 m. At the end of the Phase II the length was extended to 500 m.

2. PHASE I1 TECHNICAL OBJECTIVES

The main Phase II Objectives were:

- Develop a substrate with reduced ac loss; strength needs to be at par with Ni-5W or, more desirable, significantly enhanced.
- Develop a conductor with a transposed filamentary structure without twisting the conductor, and demonstrate significantly reduced hysteretic losses.

2.1 RABiTS substrates

Most suitable substrate alloys for the RABiTS process are modest in strength and often weakly magnetic. Development efforts worldwide aim at enhanced strength and reduced magnetism. The magnetism influences losses in two ways: it leads to ferromagnetic losses and its permeability enhances losses in the YBCO layer. A reduction in both is beneficial for the ac loss in the 2G HTS. Note that fully non-magnetic alloys such as Ni-13Cr texture well but the presence of Cr poses problems with buffer layer deposition.

2.2 Filament patterning designs

The “Flattened Barber Pole” concept aims at global AC loss reduction by inducing a “twist” in filaments without actually twisting the wire. The concept uses two 2G

conductors, each having a Ag cap layer and a partial coating with a thin insulating layer as shown in Fig. 1, top. Each conductor is then laser-patterned as shown in Fig. 1, bottom. The essential feature of this design is the presence of two parallel conducting stripes on the cap surface, which are left uncovered by the insulating layer. The two insulated faces of the patterned 2G sections are then bonded together so that an electrical connection is formed only between the overlapping end areas of matching which cross the conducting stripes. The width and the position of the conducting stripes can be chosen in such a way that each filament in one 2G layer is electrically connected with only two filaments in the other 2G layer as shown in Fig. 2.

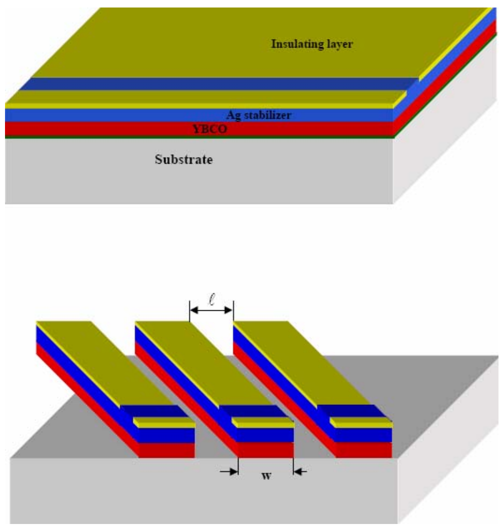


Figure 1. Top: 2G with Ag cap and insulating layers before patterning. Bottom: 2G with slanted filaments after laser patterning

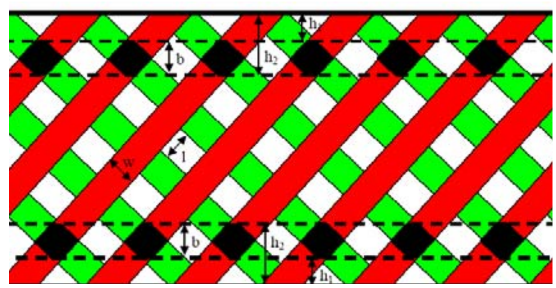


Figure 2. Zig-zag pattern formed by slanted filaments in the top (red) and bottom (green) conductors. Filament-filament contact is in dashed regions only.

This geometry provides the same effective filament decoupling as does twisting in LTS multifilamentary wires. Current transport between different filaments which belong to the same zigzag current path will see some electrical contact resistance between crossed filaments at the conducting end areas, which was expected to be kept to acceptable limits.

3. PHASE II WORK PLAN

The Phase II Work Plan had the following Tasks:

1. Evaluate Powder Metallurgy (PM) NiW foil manufacture.
   - Evaluate low C PM Ni-5W against benchmark ingot Ni-5W.
   - Evaluate texture development in Ni-9W.
   - Other approaches to RABiTS substrates with reduced ac loss
2. Fabricate unlaminated 2GHTS with state of the art J_c.
3. Develop accurate laser patterning
4. Measure performance of filamentized conductor.
5. Manufacture short lengths of patterned and in-plane twisted filaments.

6. Measure ac loss as a function of pattern geometry, filament spacing, and filament width (UW).
7. Model ac loss behavior as a function of pattern geometry (UW).
8. Reporting

4. EXECUTION OF WORK PLAN

4.1 Evaluate Powder Metallurgy (PM) NiW foil manufacture

4.1.1 Evaluate low C PM Ni-5W against benchmark ingot Ni-5W.

As discussed before, substrates made using PM Ni-5W, with 150-250 ppm C, often resulted in a retained roll texture and a 100% cube texture did not develop during the texture anneal. A low C test-batch of Ni-5W strip was therefore ordered which was virtually free of C but had a 200-400 ppm O in the matrix. The oxide inclusions again led to a retained roll texture. Since then process developments at the vendor have steadily improved strip quality but not to a full acceptance for actual substrate use yet.

4.1.2 Evaluate texture development in Ni-9W.

Ni-9W is an ideal composition in terms of strength (σ_{yield}= 400 MPa at 77 K) and magnetic properties (T_{Curie} ~0 K). However, both AMSC and independent work at IFW in Germany noted a transition above Ni-5W: the texture at 7 and 9 at% W is no longer a pure cube texture but shows an increasing presence of the brass texture. Both PM- and ingot Ni-9.3W- based substrates showed a 50% cube texture, insufficient for 2G HTS manufacture. This high W compositional range was therefore abandoned.

4.1.3 Other approaches to RABiTS substrates with reduced ac loss

Solid solution NiW(X) alloys

Several other approaches were explored. The first one was a ternary alloy approach, and the second one a bi-metal approach. In the ternary alloy approach a small ingot caster was used for the manufacture of 200 gram ingots. These were homogenized and hot rolled to strip and cold rolled to foil. The base alloy composition was Ni-5W, to which 1 or 2 at% Si, V, Nb, Al, or Be were added. Nb, Al and Be were eliminated early: only random textures resulted from these additions. Si and V did show cube textures but not as fully developed as the base Ni-5W alloy. To understand the results, the lattice constant was plotted versus the resulting cube texture for these alloys, including earlier made NiCr and NiCrW alloy substrates. The result is shown in Figure 3, left. On the right the relation between the lattice constant and yield stress in some annealed alloy substrates is shown. Up to a_o=3.555A all compositions (with the exception of Ni-5Si) show a sharp cube texture. Ni-7Cr-4W and Ni-5W-1Si slightly exceed this 3.555 A value and start showing deviations from the >95% cube texture content. Ni-5W-2V, Ni-7W and Ni-9.3W show increasingly deviating textures, with Ni-9.3W having less than 50% cube texture. All in all, the strongest alloy with optimal texture remains Ni-5at%W. With reducing lattice constant the alloy is cube textured but often significantly weaker. This is in particular the case for the Cr-bearing alloys.

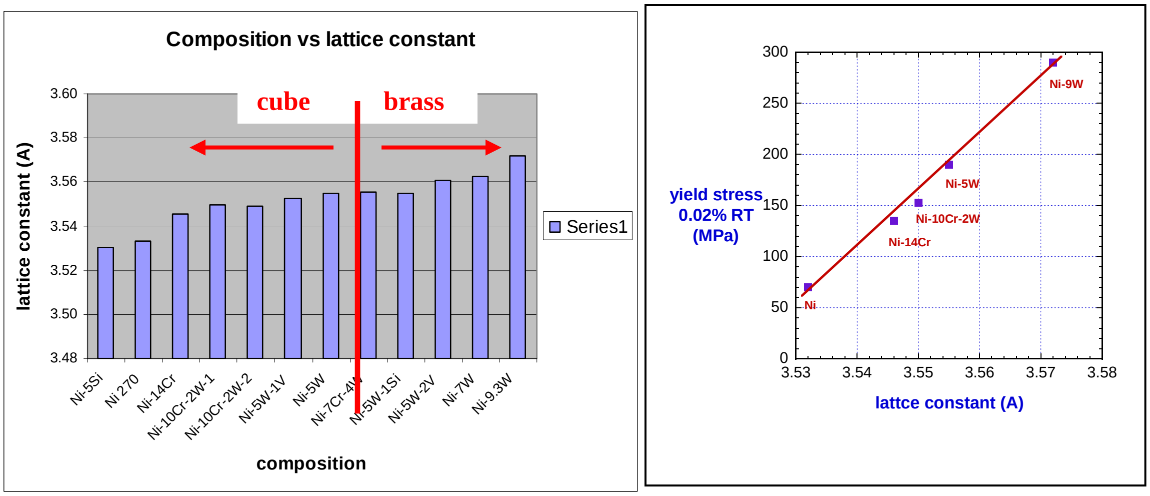
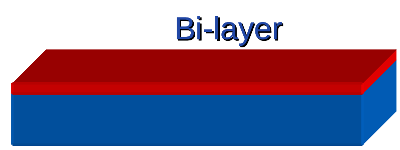


Figure 3. Lattice constant as a function of composition in NiW and NiWX alloys , left, and yield strength as a function of lattice constant, right

<u>Bi-or Tri- layer substrates</u>

In the bi-or tri-layer concept the sheath is Ni-4W or Ni-5W, which textures well, and can be used as a deposition surface. The core does not need to texture and is only used for its strength. A requirement is that the core does not impede texture formation in the sheath, or poison the buffer and YBCO layer with rapidly diffusing elements.

Figure 4. Sketch of a bi-layer substrate. The Ni-4W top provides the texture and the Ni-9W core (bottom) provides strength and reduced magnetism.



Initial efforts used a pure Ni sheath and a Ni-9.3 at% W core, using a 20/80 ratio for thickness. This did not work well: the Ni-9W penetrated the Ni sheath and texture development was poor. In a second effort a Ni-4W sheath was selected, in combination with a Ni-9W core. This approach worked much better. The sheath textured as expected, and the resulting strength (260 MPa) followed a rule of mixtures, based on a 20% sheath with a yield stress of 150 MPa for Ni-4W and the 290 MPa for the remaining Ni-9W.

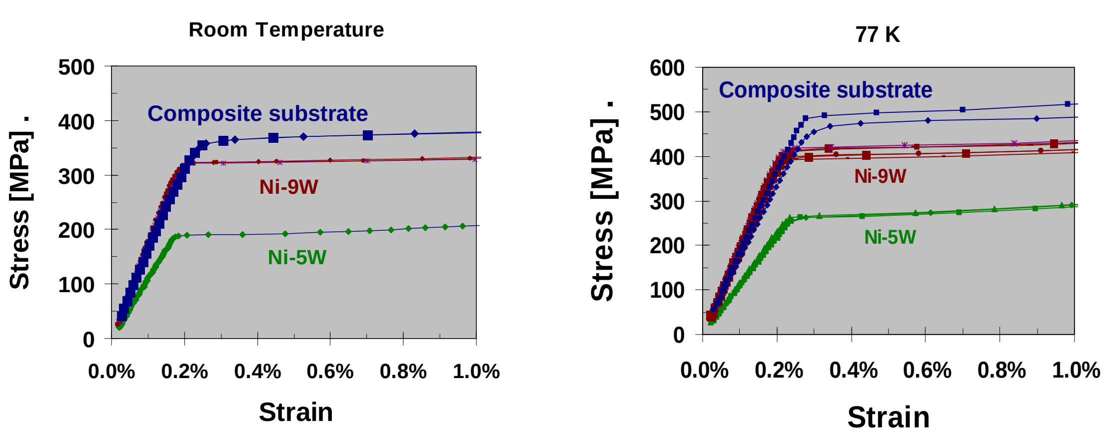


Figure 5. Stress-strain for a composite (bi-layer) substrate, Ni-4W top, at RT and 77 K.

Since then development efforts focused at ways to enhance the strength of the core material further. A core material was developed which enhanced strength to well over that of Ni-9W, as can be seen in Figure 5. At 77 K the 0.02% yield stress was 475 MPa.

Magnetic measurements were done at NRL, by J. Claassen. These showed that the relative permeability was strongly reduced, as can be seen in Figure 6. On the other hand, the ferromagnetic loss was comparable to that of Ni-5W. The B-H curves are shown in Figure 7. While the remanent magnetization is greatly reduced, from 0.12T in Ni-5W to 0.02 T in the bi-layer substrate**,** the coercive field is increased, from the low 140 A/m in NiW to around 500 A/m in the bi-layer, see Figure 9.

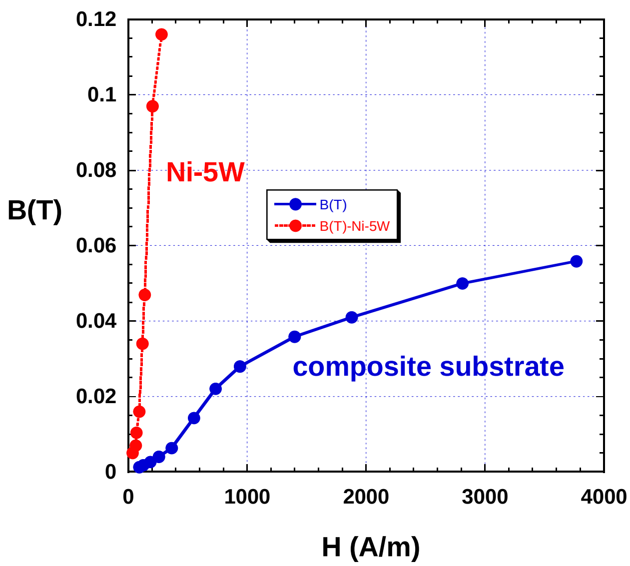


Figure 6. Magnetic measurements at 77 K, B// substrate surface. Regular Ni-5W and composite bi-later substrate (see Figure 7).

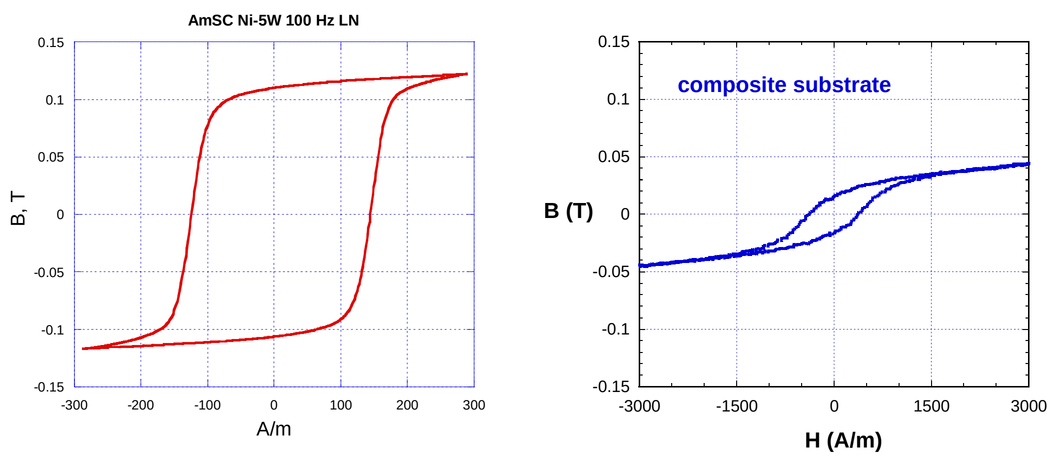


Figure 7. B-H loops for Ni-5W (left) and composite substrate (right) both at 77 K. Enclosed surface area is comparable. Remanent magnetization is higher in Ni-5W, but coercive field is higher in bi-layer substrate.

Superconducting performance was demonstrated in a short length of conductor, made using the bi-layer substrate from figures 7 and 8. A performance of 300A/cm width was demonstrated (~1 μm thick YBCO layer) showing that the bi-layer approach is suitable for 2G HTS manufacture, and shows no interference with buffer or YBCO quality.

4.2. Fabricate unlaminated 2G HTS with state of the art J_c.

As explained in the Introduction, present 2G HTS manufacture uses a process width of 40 mm. Sections of this conductor, after Ag deposition and oxygen annealing, but before slitting to the usual 4 mm width, were sent to UW for the Flattened Barber Pole development. Performance was in the 200-220 A/cm range. Un-laminated 4 mm wide insert HTS was used for striation efforts.

4.3 Develop accurate laser patterning and measure performance in patterned conductor

Laser patterning for AMSC was done by Mound Laser Photonics (Miamisburg OH). AMSC used conductors which were slit to 4 mm and then striated. Three 140 mm long sections were cut form a 74 A 2G conductor - with Ag layer, no further copper stabilizer. These lengths were laser striated over a 100 mm length with the following details, see also Figure 8:

- Striation was into 7 filaments, each being 450 μm wide and 100 mm long.
- Laser-affected path was around 50 μm.
- The two ~150-200 μm wide edges (Figure 8) were isolated from the rest as they had some slitting damage.
- All samples had common 20 mm long current pads
- Samples were measured as patterned and with an additional 1.5 μm Ag+O2 anneal, at AMSC (SF) and at ORNL (77 K, 0-1.5 Tesla perpendicular field).

The max anticipated current was based on the anticipated "ideal" current in the 74 A conductor assuming that no slitting damage had occurred:

- I_{ideal}= 1.1 x 74A= 81.4 A.
- I_c as striated is calculated assuming a 50 μm wide laser path in seven filaments:
- I_c= I_{ideal} x (7 x 0.45)/4.1)= **62.5 A**

Table 1 shows the I_c (77 K, SF) for the three samples. Number 2 and 3 are very similar, and show an I_c retention of around 95% (or 80% if compared without considering loss in real estate). Sample #1 shows a lower value. At present we do not have the capability to test this sample and see if the lower I_c is related to a flaw or to a true percolation problem somewhere along the length.

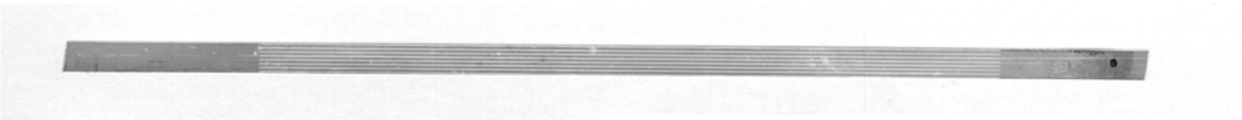


Figure 8. Striated conductor with 450 μm wide filaments and 200 mm wide edge. Top: 140x4 mm striared conductor. Right: enlarged view near edge.

Edge was isolated in measurements in all three samples.

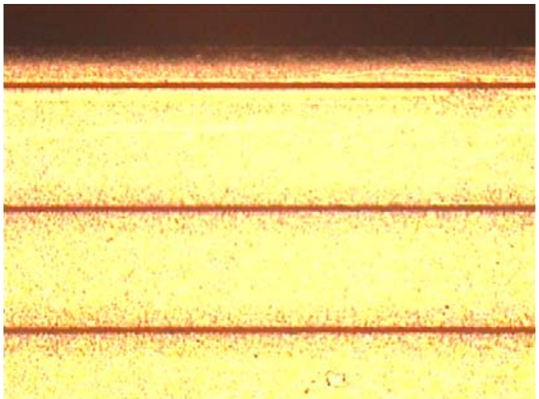


Table 1. I_c at 77 K, SF for three striated conductors

wire #	2nd 1.5 um Ag	2nd O2-anneal	Ic (A)
1	no	no	45.1
2	yes	yes	59.2
3	yes	no	59.7

Two samples were measured in a perpendicular field at 77 K (ORNL). The blue sample (#2 in Table 2) showed a field dependence comparable to un-striated samples. I_c retention at 1 Tesla was around 17%. The #1 sample which showed a lower I_c at 77 K, SF showed comparable performance at 1.5 T, see Figure 9.

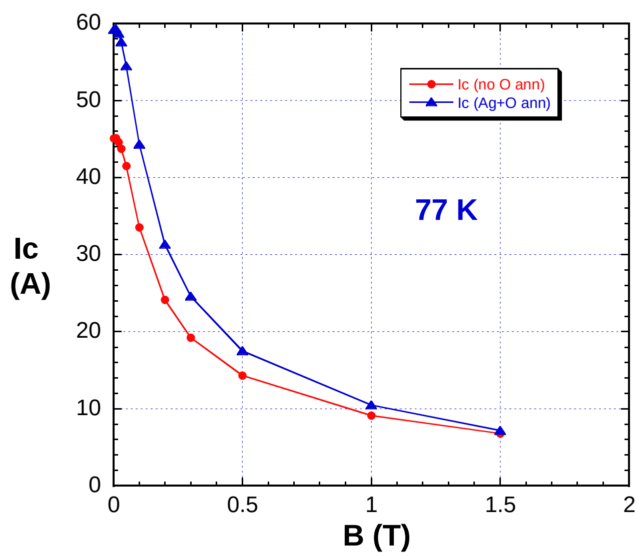


Figure 9. I_c in two striated conductors at 77 K, and B_{perp} of 0-1.5 T.

4.4 FLATTENED BARBER POLE CONCEPT (UW/FSU)

The main goals for this part of the project were: 1. Develop filament patterning of AMSC 2G and insulator deposition techniques. 2. Investigate the optimum heat treatment for thermal bonding of the 2G-2G sections to minimize the interface contact resistance and provide the best mechanical strength of the bonded conductor.

4.4.1. Filament patterning

Wet chemical etching was used to form the desired transposed filament pattern. To make the pattern design easy and reproducible, a LabView based program was written in which all pattern parameters can be varied. We made masks for 11 mm × 5 mm rectangular samples with different filament widths: 25, 50, 100, 200, 300, 400, 500 μm. The filament spacing was equal to the filament width and the inclination angle was 45^o for all patterns. Two sets of alignment marks were also produced on each pattern, as

shown in Figure 10. The Quartz 4 inches × 4 inches photo-mask was made by Microtronics, Inc according to our AutoCad 2004 sketch.

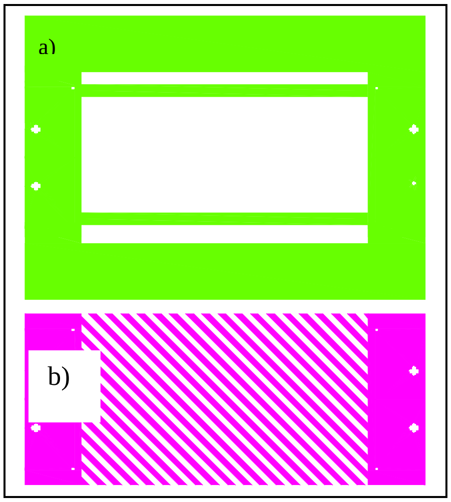


Figure 10. Pattern for the formation of contact areas (a); Pattern for the filaments formation (b).

The photo-mask in Figure 10 was used to make 6 pilot (3 pares) specimens with 200 μm filament width. A PMMA AZ1518 photo-resist was spun on at 6000 RPM. The resist was soft-baked and the patterned filaments were exposed for 40 sec in the mask aligner (Karl Suss Model 505) through the pattern shown in Figure 10 (b). The resist was developed and hard baked to ensure stability to the silver etching solution. To form the pattern the silver was removed in PMMA open areas using the solution of NH_4OH:H_2O_2:H_2O) (Figure 11a). Then YBCO was dissolved by 1% HNO_3 in water (Figure 11 (b)).

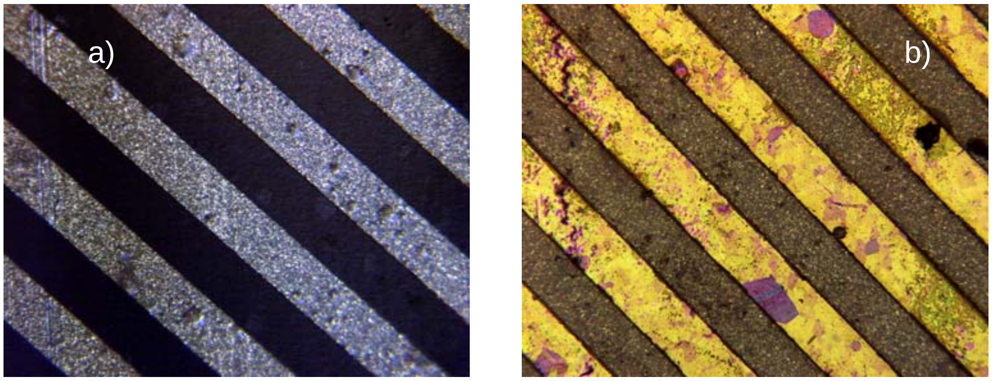


Figure 11 Fragment of the specimens at different preparation stages: a) after the silver is removed; b) after YBCO is removed with the buffer layer open.

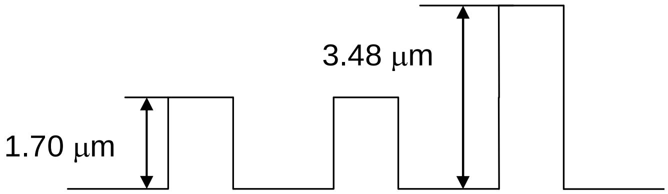


Figure 12. Sketch of the profile near the contact area corrected for the specimen curvature.

As shown in Figure 12, the height of the silver clad filaments is 1.7 μm, while the height of the 222 μm wide contact strips is about 3.5 μm. The silver clad filaments will be covered with a photo-resist dielectric layer up to 1.8 μm thick. Figure 13 shows the patterned sample before the last photolithographic stage – covering with dielectric.

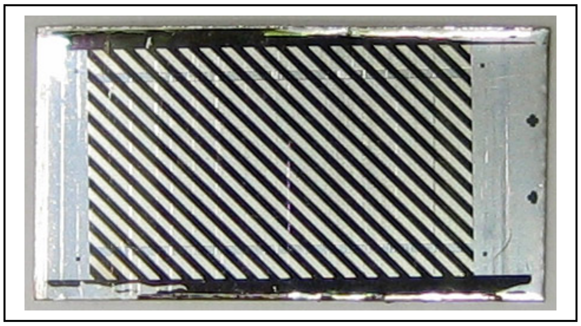


Figure 13. Macro view of sample with 200 um filaments. Contact areas are just visible as two horizontal stripes of the silver steps

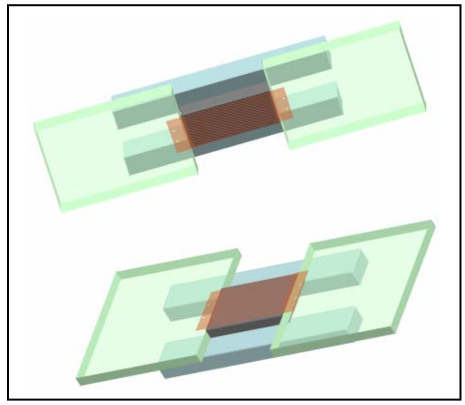


Figure 14. Sketch of the sample arrangement before alignment.

To align two specimens before thermal bonding the following procedure were used:

- Pattern with alignment marks during the last photolithography stage (covering with insulator) on two microscope cover glasses.
- Glue glasses to 316ss sample holder with windows near alignment marks, Figure14.

An aligner was designed and assembled to be used with a stereo microscope to align specimens, based on a X-Y-Z-rotation stage (Edmund Industrial Optic X-Y stage, straight line accuracy 2 μm/ 25 μm of travel, see Figure 15). Also designed were elongated Helmholtz cooper coils with a small 8-shaped pick-up coil, also sued for ac loss measurements in liquid nitrogen. The coil produced 40 mT peak magnetic field.

Figure 15. Aligner.

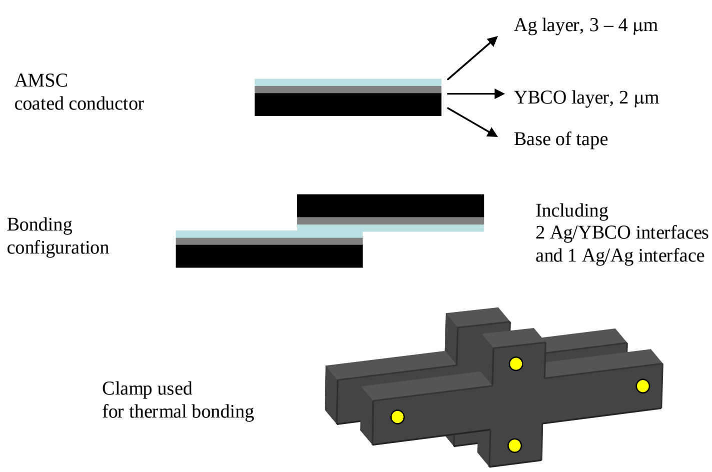


Figure 16 Sample preparation.

4.4.2. Thermal bonding and optimizing the contact resistance.
The contact resistance between two CC samples bonded at different temperatures for and durations was measured in the superconducting state at 77K, Figure 17

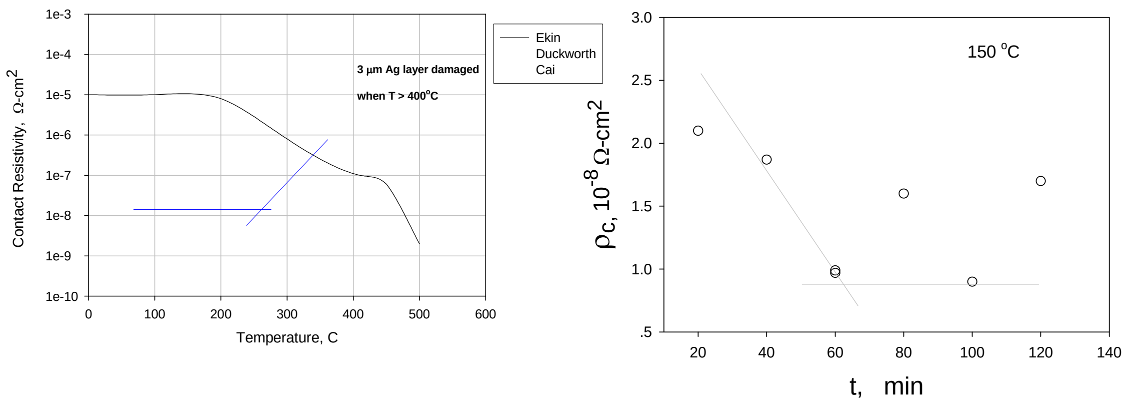


Figure 17. Ag-YBCO interface resistance in literature and measured versus baking time.

Also investigated were the optimum baking time which provides the maximum strength to withstand disconnecting the bonded conductors, and contact resistance versus bonding area, Figures 18 and 19. Thermal bonding was at 150C. Finally, optimal bonding time was determined for minimal resistance. The minimum time for lowest ρ_c is ~ 60 minutes.

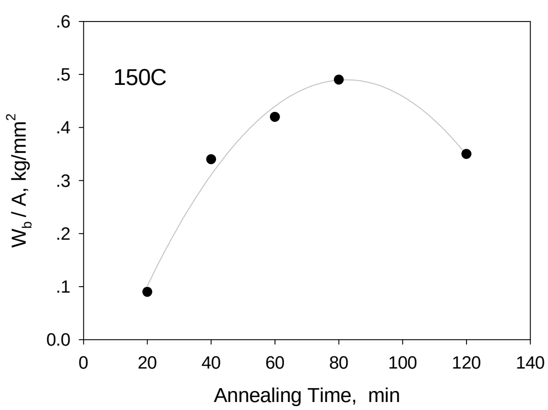


Figure 20. Mechanical strength of bonding Versus bonding time at 150°C

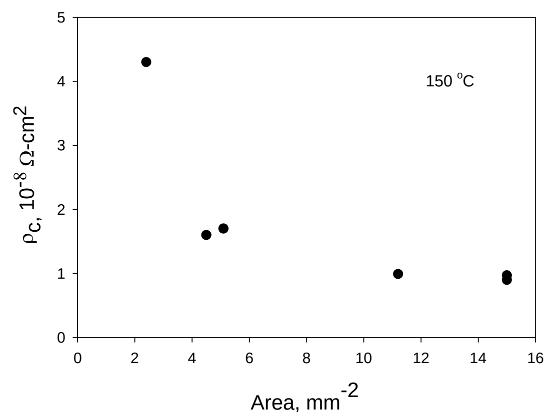


Figure 21. Effect of bonding area on contact resistance

Conclusions on realization of Flattened Barber Pole concept

1. The optimization of bonding between coated conductors has been studied with 4 varying process parameters: temperature, time, bonding area, and pressure.
2. The total contact resistivity of the bonding can be reduced down to 5 X 10^{-9} Ω-cm^2.
3. Bonding ***resistivity*** decreases as the bonding area decrease, perhaps due to a non-uniform contacting of the bonding or by a large current transfer distance
4. Proper pressure may improve the electrical contact between Ag and YBCO of the same 2G, and Ag and Ag of two thermally bonded 2G HTS.

4.4.3 AC loss characterization and demonstration of Barber pole concept

The following results were taken from a paper which has recently been submitted to Applied Physics Letters:

Dmytro Abraimov1, Alex Gurevich, Anatolii Polyanskii, X.Y. Cai, Aixia Xu, Sastry Pamidi, David Larbalestier, and C.L.H. Thieme, *"Significant reduction of AC losses in YBCO patterned coated conductors with transposed filaments"*.

Three samples were patterned as described above and hysteretic losses were measured and compared against unpatterned 2G HTS. The magnetization ac loss power $Q(H)$ at 77 K on bonded patterned samples and an un-patterned control two-sided CC of the same dimensions were measured at AC fields up to 100mT at frequencies from 10 Hz to 400 Hz, applied with B//c. The paper's figures 2 and 3 present the most significant findings:

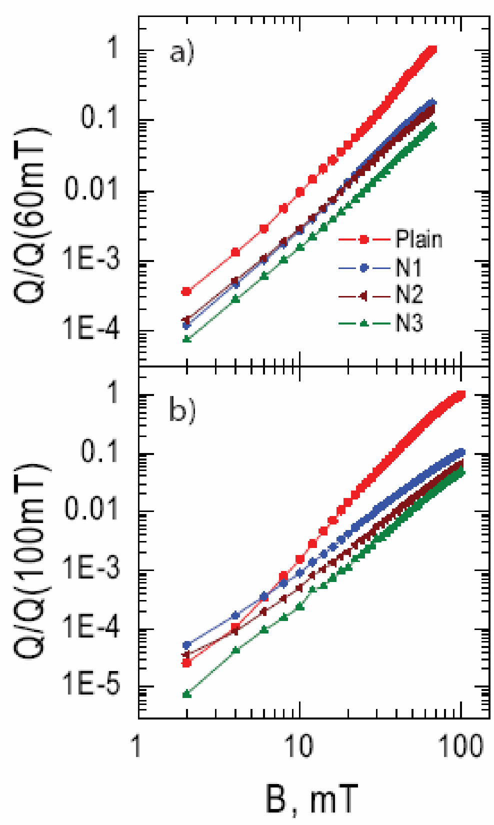


FIG. 2: Losses normalized to the maximum Q of the controlled sample for three patterned samples at 400 Hz (a) and 55 Hz (b).

Fig. 2 shows the field dependencies of $Q(H)$ for samples N1, N2, and N3, and the control sample. AC losses of patterned samples at high fields drop by ~10-15 times as compared to the control sample for all frequencies from 10 to 400 Hz.

For decoupled filaments at $B > \mu_o Jcd/\pi$, it is estimated that:

$Q_{contr}=Q_{pattern} = (W/w)^2/N$, and
$Q_{contr}=Q_{pattern}$ approaches 13 for W = 4 mm (the width of the 2G), w = 0.5mm (the filament width) and N = 5 filaments across the sample.

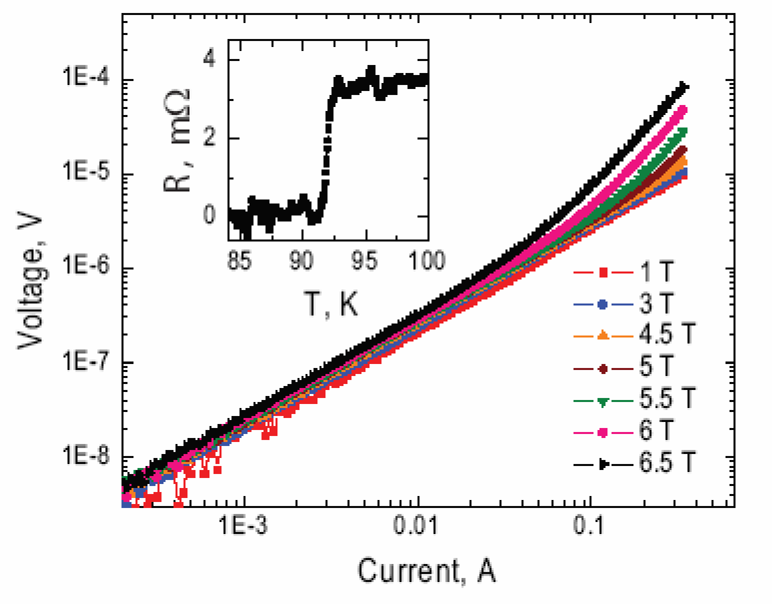


FIG. 3: $V - I$ characteristics of patterned sample N1. The resistive transition at $H = 0$ is shown in the inset.

This estimate of loss reduction is consistent with the data shown in Fig. 3, indicating no degradation of local J_c in the filaments during patterning and thermal bonding.

5. CONCLUSIONS

This Phase II STTR Project aimed at the development of a substrate with reduced ac loss, coupled with enhanced strength, and the development of a conductor with a transposed filamentary structure without twisting the conductor. These goals have been met.

<u>Substrate</u>:
The viability of a multi-layer substrate was demonstrated in short lengths. A bi-layer substrate with a Ni-4W sheath and strong core demonstrated:

- A reduction in permeability of a factor of 7(ferromagnetic loss remained comparable to that of Ni-5W0
- Strength nearly doubled over that of the baseline Ni-5W.
- A fully coated conductor with this substrate demonstrated 300 A/cm-width

<u>Barber-pole concept for ac loss reduction</u>

- The concept, while complex in nature, could be realized as planned, with minimal losses in the bonded areas.
- AC losses of patterned samples at high fields drop by ~10-15 times as compared to the control sample for all frequencies from 10 to 400 Hz.

Presentations and publications

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Cees Thieme, Dawood Aized, "Reduced ac loss and stability considerations in AMSC Second Generation Superconductors ", Stanford Wisconsin Workshop on Coated Conductors USAF Program Review, Palo Alto CA, April 24-25, 2006.

C.L.H. Thieme, M.W. Rupich, X. Li, W. Zhang, T. Y. Huang, T. Kodenkandath, N. Nguyen, D. Aized, J. Voccio, and D.T. VerebelyiDevelopment of Second Generation HTS Wire at American Superconductor", 2007 MRS Spring Meeting San Francisco CA, April 10 2007.

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